Acta Cryst. (1967). **23**, 384

The Crystal and Molecular Structure of Nickel Bis(dithiocarbamate)

By Giovanna Fava Gasparri, Mario Nardelli and Angiola Villa
Istituto di Strutturistica Chimica della Università degli Studi, Parma, Italy

(*Received* 30 *December* 1966)

The crystal structure of nickel bis(dithiocarbamate), $Ni(S_2CNH_2)_2$, has been determined and refined using three-dimensional Fourier methods (final $R=11{\cdot}2\%$). The monoclinic ($P2_1/c$) unit cell of dimensions $a=8{\cdot}96(2)$, $b=9{\cdot}825(5)$, $c=11{\cdot}28(2)$ Å, $\beta=129°16'(7')$, contains four molecules in which the nickel atom coordinates to four sulphur atoms (mean Ni–S distance, $2{\cdot}21_5$ Å) in a nearly centrosymmetrical planar arrangement. Significant deviations from planarity in the coordination plane are observed which can be explained by packing and hydrogen bonding interactions concerning sulphur atoms and NH_2 groups.

Introduction

In attempting to prepare metal complexes of thiouram derivatives, some metal dithiocarbamates were obtained: these were considered interesting for an X-ray analysis, as no structural determination has yet been carried out on unsubstituted dithiocarbamates. Previous work has been restricted to the metal complexes of *N*-alkyldithiocarbamates (Peyronel, 1941; Alderman, Owston & Rowe, 1962; Bonamico, Dessy, Mariani, Vaciago & Zambonelli, 1965; Bonamico, Dessy, Mugnoli, Vaciago & Zambonelli, 1965; Bonamico, Mazzone, Vaciago & Zambonelli, 1965; Colapietro, Domenicano, Vaciago & Zambonelli, 1966; Klug, 1966; Peyronel & Pignedoli, 1966; Shugam & Agre, 1966; Zvonkova, Poveteva, Vozzhennikov, Glushkova, Yakovenko & Chvatkina, 1966). X-ray studies on unsubstituted dithiocarbamates are useful for studying the influences of the substituents on the geometry of coordination, on the nature of the bonds, on packing and other structural features, and for comparing all these features with the corresponding substituted derivatives.

In the present paper the results of a three-dimensional X-ray analysis of nickel bis(dithiocarbamate), $Ni(S_2CNH_2)_2$, are reported.

Experimental

Nickel bis(dithiocarbamate) was prepared by mixing nickel(II) chloride and ammonium dithiocarbamate in aqueous solution, and crystals suitable for X-ray analysis were grown from ethanol as short green-black monoclinic prisms. Cell constants, determined from rotation and Weissenberg photographs and refined by a least-squares procedure on powder diffractometer data (Cu $K\alpha$, $\lambda=1{\cdot}5418$ Å), are as follows:

$Ni(S_2CNH_2)_2$. $M=243{\cdot}0$
$a=8{\cdot}962\pm0{\cdot}018$, $b=9{\cdot}825\pm0{\cdot}005$, $c=11{\cdot}280\pm0{\cdot}019$Å, $\beta=129°16'\pm7'$, $V=769{\cdot}0$ Å^3, $Z=4$, $D_x=2{\cdot}098$, $D_m=2{\cdot}09$ g.cm^{-3} (flotation). $\mu=128{\cdot}2$ cm^{-1} (Cu $K\alpha$). $F(000)=480$.
Space group: $P2_1/c$ from systematic absences.

Three-dimensional intensity data were determined photometrically on integrated equi-inclination Weissenberg photographs (multiple film technique; Cu $K\alpha$) taken up to the ninth and tenth layers along [100] and [010] respectively. Of the 1755 possible independent reflexions within the limiting sphere, 1292 were strong enough to be observed. The shape of the spots of non-equatorial layers was taken into account following Phillips (1956), and to correct for absorption the samples were considered as cylindrical ($\bar{r}=0{\cdot}04$ cm) and spherical ($\bar{r}=0{\cdot}03$ cm) for reflexions taken around [100] and [010] respectively. After correction for Lorentz and polarization factors, the intensities were placed on the same relative scale following Rollett & Sparks (1960); the absolute scale was then established for all the three-dimensional data, by Wilson's method.

Structure analysis and refinement

The structure was solved by the heavy-atom method, starting from Patterson coordinates for Ni, and refined by several cycles of Booth's differential synthesis with the anisotropic thermal parameters which were determined using the second derivatives of the electron-density from differential synthesis (Nardelli & Fava, 1960). The final residual error indices (R for observed reflexions only, R' assuming $F_o=\frac{1}{2}F_{\min}$ when $F_c\geq F_{\min}$ for unobserved reflexions, multiplicities not considered) were $R=11{\cdot}2\%$, $R'=12{\cdot}4\%$. The introduction of hydrogen atoms in positions calculated (Table 1) to make N–H $=1{\cdot}03$ Å with trigonal hybridization for nitrogen atoms, did not change the final R indices significantly.

In Table 2 the final coordinates with e.s.d.'s (Cruickshank, 1949, 1956; the calculations were performed with computer programs taking into account the atomic anisotropy), the final anisotropic thermal parameters and the ratios $r(x)=\sigma(x)/\varepsilon(x)$ between e.s.d.'s

Table 1. *Calculated hydrogen coordinates*

	x/a	y/b	z/c
H(1)	−0·273	0·259	−0·454
H(2)	−0·356	0·191	−0·358
H(3)	0·716	0·532	0·544
H(4)	0·790	0·604	0·443

and coordinate shifts, are given. The observed and calculated values of the electron density and the second derivatives at the atomic peaks with the e.s.d.'s are compared in Table 3. The F_c values listed in Table 4 are calculated with the final parameters of Table 2 and include the contributions of hydrogen atoms, the scattering factors used being those of Thomas & Umeda (1957) for Ni^{2+}, of Dawson (1960) for S, and of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) for N and C. The standard deviations quoted in the next section are calculated from the formulae of Ahmed & Cruickshank (1953) for bond lengths and of Darlow (1960) for angles, the effects of errors in cell parameters being accounted for following Darlow & Cochran (1961).

The calculations were performed with the computer programs of Nardelli, Musatti, Domiano & Andreetti (1964, 1965).

Discussion

The coordination around each nickel atom is nearly planar and concerns four sulphur atoms of two CS_2 groups as shown in Fig. 1. From the bond distances and angles quoted in Table 5 it appears that the Ni–S distances for the four sulphur atoms are not significantly different, but are significantly shorter than the sum of Pauling's covalent radii (2·25 Å) and lie just in the middle of the range of values (2·1–2·3 Å) generally found for Ni(II) planar complexes. These distances are much shorter (~0·3 Å) than those present in octahedral Ni(II) complexes as already observed by Cavalca, Nardelli & Fava (1962) and by Lopez-Castro & Truter (1963). Considering the planar nickel complexes, the Ni–S distances in five-membered coordination rings are significantly shorter than in four-membered rings: this is evident from the values quoted in Table 6, where the difference is 0·06 Å.

Coordination around Ni is not strictly planar, the sulphur atoms being significantly out of the mean plane through them as shown in Table 7. The perfect planarity, required by symmetry conditions in other *N*-alkyl substituted nickel dithiocarbamates [*e.g.* nickel bis(diethyldithiocarbamate) and nickel bis(di-n-propyldithiocarbamate)] is now absent, probably as a consequence of NH···S interactions with adjacent molecules. As can be seen from the data in Table 7, the

Table 2. *Final atomic fractional coordinates* (× 10^4), *thermal parameters* (× 10 $Å^2$) *with e.s.d.'s and ratios* (*e.s.d.*)/(*coordinate shift*)

	x/a (σ)	y/b (σ)	z/c (σ)	B_{11} (σ)	B_{22} (σ)	B_{33} (σ)	B_{12} (σ)	B_{13} (σ)	B_{23} (σ)	$\|r(x)\|$	$\|r(y)\|$	$\|r(z)\|$
Ni	2180 (5)	3941 (2)	451 (4)	47 (4)	44 (0)	32 (4)	−1 (2)	21 (5)	0 (2)	8	4	9
S(1)	841 (8)	3821 (3)	−1994 (6)	43 (7)	47 (1)	22 (7)	−7 (3)	17 (8)	−3 (3)	13	15	19
S(2)	3652 (8)	3955 (2)	2929 (5)	46 (7)	36 (1)	23 (7)	−7 (2)	18 (8)	−2 (2)	∞	∞	14
S(3)	4848 (7)	5090 (3)	1378 (5)	37 (6)	34 (1)	24 (6)	−2 (2)	17 (7)	−1 (2)	14	6	9
S(4)	−477 (7)	2757 (3)	−495 (6)	38 (6)	41 (1)	26 (7)	−4 (3)	18 (8)	0 (3)	12	6	19
N(1)	−2538 (28)	2434 (10)	−3548 (19)	46 (22)	41 (3)	23 (23)	−12 (8)	10 (27)	−6 (8)	139	49	27
N(2)	6940 (28)	5469 (10)	4430 (18)	50 (25)	47 (4)	25 (24)	−14 (8)	15 (29)	−10 (8)	69	33	178
C(1)	−941 (29)	2950 (10)	−2185 (22)	36 (22)	28 (2)	22 (24)	−1 (9)	8 (27)	0 (9)	98	10	109
C(2)	5337 (32)	4920 (8)	3079 (23)	39 (23)	24 (3)	20 (23)	−1 (9)	11 (27)	−2 (9)	9	83	15

Table 3. *Atomic peak heights* (e.$Å^{-3}$), *curvatures* (e.$Å^{-5}$) *and e.s.d.'s*

		ϱ	$-A_{hh}$	$-A_{kk}$	$-A_{ll}$	A_{kl}	A_{hl}	A_{hk}
Ni	obs.	50·2	403	448	456	−9	264	−3
	calc.	49·8	406	447	456	−9	265	−3
S(1)	obs.	28·9	244	242	289	4	163	−20
	calc.	28·3	245	242	288	3	163	−19
S(2)	obs.	31·1	246	289	303	−1	168	−20
	calc.	30·2	247	289	303	−1	168	−20
S(3)	obs.	32·3	281	292	326	−3	188	−7
	calc.	31·3	283	292	326	−4	189	−6
S(4)	obs.	30·6	265	261	300	−2	174	−11
	calc.	29·6	266	262	301	−2	175	−11
N(1)	obs.	8·2	56	64	70	−4	33	−7
	calc.	8·2	57	64	70	−4	34	−7
N(2)	obs.	9·1	65	63	87	−4	45	−8
	calc.	8·9	67	63	88	−3	47	−8
C(1)	obs.	8·0	58	80	68	−1	35	3
	calc.	8·0	58	80	69	−1	35	3
C(2)	obs.	8·6	61	79	73	−5	41	2
	calc.	8·6	62	80	73	−5	42	2
	e.s.d.	0·3	2	3	3	2	2	1

Table 4. *Observed and calculated structure factors*

A minus sign for F_o means 'less than'.

h	k	l	$\lvert 10F_o\rvert$	$10F_c$
1	0	0	263	168
2	0	0	194	119
3	0	0	214	-207
4	0	0	453	420
5	0	0	102	-90
6	0	0	135	-125
7	0	0	424	-459
8	0	0	22-	18
9	0	0	19-	-15
1	1	0	751	-576
2	1	0	381	224
3	1	0	267	252
4	1	0	334	307
5	1	0	206	198
6	1	0	87	-82
7	1	0	121	118
8	1	0	121	141
0	2	0	670	430
1	2	0	1454	-1327
2	2	0	130	65
3	2	0	528	-502
4	2	0	194	184
5	2	0	144	-131
6	2	0	219	223
7	2	0	88	-83
8	2	0	64	78
1	3	0	1630	-1645
2	3	0	429	-352
3	3	0	97	-43
4	3	0	277	-236
5	3	0	257	-257
6	3	0	378	-386
7	3	0	37	35
8	3	0	102	95
0	4	0	336	-260
1	4	0	491	-377
2	4	0	852	837
3	4	0	116	-77
4	4	0	408	390
5	4	0	355	-331
6	4	0	206	193
7	4	0	142	131
8	4	0	17-	5
1	5	0	386	296
2	5	0	320	247
3	5	0	216	196
4	5	0	59-	60
5	5	0	213	210
6	5	0	103	90
7	5	0	28-	-6
8	5	0	21	-22
0	6	0	529	-461
1	6	0	383	-419
2	6	0	506	480
3	6	0	320	-314
4	6	0	85	78
5	6	0	249	-254
6	6	0	132	123
7	6	0	88	77
1	7	0	500	480
2	7	0	64	54
3	7	0	59	56
4	7	0	132	-142
5	7	0	42-	13
6	7	0	135	132
7	7	0	48	-49
0	8	0	616	621
1	8	0	103	-91
2	8	0	105	104
3	8	0	161	-162
4	8	0	135	145
5	8	0	45	-23
6	8	0	24-	-18
7	8	0	39-	-28
1	9	0	90	91
2	9	0	104	-103
3	9	0	63	-66
4	9	0	58	-65
5	9	0	43	31
6	9	0	33	34
0	10	0	398	414
1	10	0	83	-93
2	10	0	50-	40
3	10	0	92	-110
4	10	0	33-	37
5	10	0	20-	6
1	11	0	164	-164
2	11	0	38-	-5
3	11	0	49	58
4	11	0	21-	6
0	12	0	34-	-13
1	12	0	108	-130
2	12	0	23-	29
3	12	0	9-	-51
0	1	1	978	-941
1	1	1	124	77
1	1	$\bar{1}$	583	-474
2	1	1	471	-320
2	1	$\bar{1}$	744	579
3	1	1	450	410
3	1	$\bar{1}$	911	817
4	1	1	538	-542
4	1	$\bar{1}$	280	-239
5	1	1	173	-179
5	1	$\bar{1}$	46-	-14
6	1	1	153	145
6	1	$\bar{1}$	84	-75
7	1	1	161	168
7	1	$\bar{1}$	190	196
8	1	1	45	-42
8	1	$\bar{1}$	26-	19
9	1	$\bar{1}$	112	-102
0	2	1	66-	39
1	2	1	291	-98
1	2	$\bar{1}$	1561	1415
2	2	1	63-	-22
2	2	$\bar{1}$	1182	1049
3	2	1	310	-311
3	2	$\bar{1}$	742	-689
4	2	1	320	-319
4	2	$\bar{1}$	424	-421
5	2	1	421	452
5	2	$\bar{1}$	192	-194
6	2	1	247	246
6	2	$\bar{1}$	42-	9
7	2	1	76	-75
7	2	$\bar{1}$	53	-43
8	2	1	24	-25
8	2	$\bar{1}$	204	-210
9	2	$\bar{1}$	67	-53
0	3	1	587	469
1	3	1	492	361
1	3	$\bar{1}$	953	754
2	3	1	164	-150
2	3	$\bar{1}$	741	-586
3	3	1	216	169
3	3	$\bar{1}$	130	74
4	3	1	59-	47
4	3	$\bar{1}$	56-	17
5	3	1	173	167
5	3	$\bar{1}$	76	67
6	3	1	217	-214
6	3	$\bar{1}$	42-	49
7	3	1	28-	-14
7	3	$\bar{1}$	54	-51
8	3	1	79	-87
8	3	$\bar{1}$	43	-41
9	3	$\bar{1}$	29-	36
0	4	1	381	273
1	4	1	322	279
1	4	$\bar{1}$	241	183
2	4	1	332	275
2	4	$\bar{1}$	528	425
3	4	1	75	46
3	4	$\bar{1}$	124	-121
4	4	1	133	-137
4	4	$\bar{1}$	88	68
5	4	1	218	211
5	4	$\bar{1}$	151	139
6	4	1	142	135
6	4	$\bar{1}$	196	198
7	4	1	26	-31
7	4	$\bar{1}$	58	58
8	4	1	6-	3
8	4	$\bar{1}$	37	-28
9	4	$\bar{1}$	15-	-20
0	5	1	193	159
1	5	1	58-	-30
1	5	$\bar{1}$	500	382
2	5	1	211	-208
2	5	$\bar{1}$	1137	-1192
3	5	1	67	43
3	5	$\bar{1}$	537	-534
4	5	1	273	271
4	5	$\bar{1}$	103	-97
5	5	1	346	353
5	5	$\bar{1}$	49-	44
6	5	1	225	-218
6	5	$\bar{1}$	42-	15
7	5	1	22-	2
7	5	$\bar{1}$	54	-56
8	5	$\bar{1}$	18-	-7
0	6	1	341	-247
1	6	1	284	-244
1	6	$\bar{1}$	821	-689
2	6	1	217	-191
2	6	$\bar{1}$	391	-361
3	6	1	56-	-5
3	6	$\bar{1}$	163	152
4	6	1	60	-41
4	6	$\bar{1}$	59-	-19
5	6	1	279	-285
5	6	$\bar{1}$	114	-133
6	6	1	168	-164
6	6	$\bar{1}$	59	-57
7	6	1	17-	-2
7	6	$\bar{1}$	88	-88
8	6	$\bar{1}$	79	84
0	7	1	84	43
1	7	1	197	171
1	7	$\bar{1}$	426	358
2	7	1	124	-107
2	7	$\bar{1}$	172	147
3	7	1	54-	-5
3	7	$\bar{1}$	270	273
4	7	1	125	-114
4	7	$\bar{1}$	111	118
5	7	1	73	74
5	7	$\bar{1}$	102	106
6	7	1	60	-72
6	7	$\bar{1}$	71	-60
7	7	$\bar{1}$	24-	-21
8	7	$\bar{1}$	5-	-64
0	8	1	154	-139
1	8	1	73	59
1	8	$\bar{1}$	432	-405
2	8	1	128	109
2	8	$\bar{1}$	159	-157
3	8	1	180	213
3	8	$\bar{1}$	248	256
4	8	1	161	172
4	8	$\bar{1}$	166	188
5	8	1	67	-64
5	8	$\bar{1}$	50	52
6	8	1	43	-42
6	8	$\bar{1}$	93	91
7	8	$\bar{1}$	18-	-8
0	9	1	405	-418
1	9	1	52-	-6
1	9	$\bar{1}$	261	-253
2	9	1	123	-114
2	9	$\bar{1}$	161	165
3	9	1	78	78
3	9	$\bar{1}$	102	102
4	9	1	164	-183
4	9	$\bar{1}$	143	-156
5	9	1	42	-51
5	9	$\bar{1}$	31	31
6	9	$\bar{1}$	40	-39
0	10	1	60-	-86
1	10	1	58	-60
1	10	$\bar{1}$	108	103
2	10	1	84	-90
2	10	$\bar{1}$	107	103
3	10	1	48	-56
3	10	$\bar{1}$	98	-107
4	10	1	60	-59
4	10	$\bar{1}$	55	-54
5	10	1	9-	36
5	10	$\bar{1}$	111	-139
6	10	$\bar{1}$	9-	-27
0	11	1	48-	-23
1	11	1	140	159
1	11	$\bar{1}$	37-	21
2	11	1	37	33
2	11	$\bar{1}$	104	105
3	11	1	54	75
3	11	$\bar{1}$	88	97
4	11	1	7-	-39
4	11	$\bar{1}$	37	-46
5	11	$\bar{1}$	7-	43
0	12	1	53	52
1	12	1	53	58
1	12	$\bar{1}$	103	113
2	12	1	18-	25
2	12	$\bar{1}$	104	121
3	12	$\bar{1}$	64	-95
0	0	2	66-	35
1	0	2	355	215
1	0	$\bar{2}$	958	816
2	0	2	755	-733
2	0	$\bar{2}$	595	-654
3	0	2	55-	8
3	0	$\bar{2}$	445	-336
4	0	2	622	688
4	0	$\bar{2}$	587	462
5	0	2	47	48
5	0	$\bar{2}$	832	859
6	0	2	37-	-7
6	0	$\bar{2}$	39-	25
7	0	2	149	-136
7	0	$\bar{2}$	242	229
8	0	$\bar{2}$	182	-179
9	0	$\bar{2}$	80	78
0	1	2	302	214
1	1	2	204	107
1	1	$\bar{2}$	631	-550
2	1	2	612	541
2	1	$\bar{2}$	1099	-964
3	1	2	694	751
3	1	$\bar{2}$	683	551
4	1	2	161	142
4	1	$\bar{2}$	940	906
5	1	2	99	-97
5	1	$\bar{2}$	211	172
6	1	2	58	58
6	1	$\bar{2}$	121	97
7	1	2	24-	6
7	1	$\bar{2}$	143	136
8	1	$\bar{2}$	135	129
9	1	$\bar{2}$	108	98
0	2	2	373	-227
1	2	2	402	-278
1	2	$\bar{2}$	490	297
2	2	2	344	-291
2	2	$\bar{2}$	1227	-1032
3	2	2	107	106
3	2	$\bar{2}$	617	-502
4	2	2	202	191
4	2	$\bar{2}$	484	-392
5	2	2	63	-59
5	2	$\bar{2}$	135	128
6	2	2	107	100
6	2	$\bar{2}$	275	-283
7	2	2	23-	20
7	2	$\bar{2}$	90	89
8	2	$\bar{2}$	87	-76
9	2	$\bar{2}$	127	114
0	3	2	72-	-76
1	3	2	640	-539
1	3	$\bar{2}$	147	143
2	3	2	129	-96
2	3	$\bar{2}$	670	-543
3	3	2	488	476
3	3	$\bar{2}$	620	526
4	3	2	59-	22
4	3	$\bar{2}$	567	551
5	3	2	331	-339
5	3	$\bar{2}$	173	-154
6	3	2	109	-104
6	3	$\bar{2}$	196	-191
7	3	2	82	-81
7	3	$\bar{2}$	163	-166
8	3	$\bar{2}$	98	-99
9	3	$\bar{2}$	43-	19
0	4	2	178	78
1	4	2	373	301
1	4	$\bar{2}$	229	176
2	4	2	573	507
2	4	$\bar{2}$	573	471
3	4	2	109	-87
3	4	$\bar{2}$	1160	1406
4	4	2	482	-499
4	4	$\bar{2}$	227	-206
5	4	2	169	-158
5	4	$\bar{2}$	152	-152
6	4	2	43	29
6	4	$\bar{2}$	127	-125
7	4	2	71	-77
7	4	$\bar{2}$	113	111
8	4	$\bar{2}$	66	-61
9	4	$\bar{2}$	43	-40
0	5	2	74-	-12
1	5	2	84	-54
1	5	$\bar{2}$	98	-61
2	5	2	299	250
2	5	$\bar{2}$	194	-143
3	5	2	112	-104
3	5	$\bar{2}$	55-	36
4	5	2	88	66
4	5	$\bar{2}$	149	-140
5	5	2	98	91
5	5	$\bar{2}$	73	39
6	5	2	125	124
6	5	$\bar{2}$	67	55
7	5	2	13-	21
7	5	$\bar{2}$	116	115
8	5	$\bar{2}$	60	55
9	5	$\bar{2}$	22-	43
0	6	2	291	-242
1	6	2	132	-102
1	6	$\bar{2}$	130	-117
2	6	2	218	191
2	6	$\bar{2}$	217	-165
3	6	2	73	71
3	6	$\bar{2}$	394	384
4	6	2	183	-183
4	6	$\bar{2}$	298	-293
5	6	2	67	-55
5	6	$\bar{2}$	78	-72
6	6	2	90	90
6	6	$\bar{2}$	116	-120
7	6	$\bar{2}$	54	56
8	6	$\bar{2}$	24	-20
0	7	2	93	73
1	7	2	71	-50
1	7	$\bar{2}$	158	145
2	7	2	68	-24
2	7	$\bar{2}$	388	332
3	7	2	527	-571
3	7	$\bar{2}$	180	-177
4	7	2	64	-42
4	7	$\bar{2}$	536	-590
5	7	2	94	85
5	7	$\bar{2}$	47-	31
6	7	2	18-	18
6	7	$\bar{2}$	45	29
7	7	$\bar{2}$	28-	2
8	7	$\bar{2}$	43	-50
0	8	2	78	-67
1	8	2	143	133
1	8	$\bar{2}$	102	82
2	8	2	129	-121
2	8	$\bar{2}$	182	-167
3	8	2	46-	5
3	8	$\bar{2}$	114	-119
4	8	2	196	204
4	8	$\bar{2}$	55-	-29
5	8	2	25-	22
5	8	$\bar{2}$	185	203
6	8	$\bar{2}$	32-	-13
7	8	$\bar{2}$	87	94
0	9	2	72	72
1	9	2	50-	-9
1	9	$\bar{2}$	65	64
2	9	2	190	199
2	9	$\bar{2}$	43	32
3	9	2	63	-71
3	9	$\bar{2}$	113	119
4	9	2	32-	30
4	9	$\bar{2}$	71	-67
5	9	2	15-	9
5	9	$\bar{2}$	58	53
6	9	$\bar{2}$	26-	-9
7	9	$\bar{2}$	14-	-2
0	10	2	59-	-65
1	10	2	42-	18
1	10	$\bar{2}$	147	140
2	10	2	175	-183
2	10	$\bar{2}$	98	-93
3	10	2	38	-37
3	10	$\bar{2}$	174	-180
4	10	2	121	157
4	10	$\bar{2}$	39-	17
5	10	$\bar{2}$	156	182
6	10	$\bar{2}$	49	-63
0	11	2	121	112
1	11	2	68	-76
1	11	$\bar{2}$	36-	26
2	11	2	38	46
2	11	$\bar{2}$	161	-169
3	11	2	67	93
3	11	$\bar{2}$	100	109
4	11	$\bar{2}$	99	112
5	11	$\bar{2}$	16-	30
0	12	2	30-	-12
1	12	2	53	55
1	12	$\bar{2}$	23-	34
2	12	$\bar{2}$	26-	3
3	12	$\bar{2}$	35	42
0	1	3	71-	89
1	1	3	283	245
1	1	$\bar{3}$	306	-257
2	1	3	831	873
2	1	$\bar{3}$	55-	89
3	1	3	218	-191
3	1	$\bar{3}$	106	-51
4	1	3	113	-118
4	1	$\bar{3}$	506	420
5	1	3	78	-72
5	1	$\bar{3}$	871	-1049
6	1	3	52	46
6	1	$\bar{3}$	36-	30
7	1	3	15-	23
7	1	$\bar{3}$	36-	-2
8	1	$\bar{3}$	135	149
9	1	$\bar{3}$	49-	-63
10	1	$\bar{3}$	27-	41
0	2	3	415	356
1	2	3	848	838
1	2	$\bar{3}$	19	-12
2	2	3	556	-512
2	2	$\bar{3}$	58-	23
3	2	3	585	-629
3	2	$\bar{3}$	113	71
4	2	3	109	-95
4	2	$\bar{3}$	837	-829
5	2	3	45	-28
5	2	$\bar{3}$	237	-206
6	2	3	29-	0
6	2	$\bar{3}$	495	517
7	2	3	13-	-62
7	2	$\bar{3}$	114	105
8	2	$\bar{3}$	45	-40
9	2	$\bar{3}$	55-	-32
10	2	$\bar{3}$	29-	-20
0	3	3	521	392
1	3	3	608	-505
1	3	$\bar{3}$	203	-123
2	3	3	242	-209
2	3	$\bar{3}$	457	317
3	3	3	56-	22
3	3	$\bar{3}$	365	-291
4	3	3	97	92
4	3	$\bar{3}$	536	474
5	3	3	43	-31
5	3	$\bar{3}$	227	211
6	3	3	27-	23
6	3	$\bar{3}$	338	334
7	3	3	8-	-47
7	3	$\bar{3}$	147	-142
8	3	$\bar{3}$	90	88
9	3	$\bar{3}$	128	-115
10	3	$\bar{3}$	21-	41
0	4	3	95	63
1	4	3	500	423
1	4	$\bar{3}$	198	-120
2	4	3	66-	-39
2	4	$\bar{3}$	43-	-3
3	4	3	108	-85
3	4	$\bar{3}$	116	85
4	4	3	93	78
4	4	$\bar{3}$	342	-296
5	4	3	97	102
5	4	$\bar{3}$	132	-125
6	4	3	24-	26
6	4	$\bar{3}$	262	261
7	4	$\bar{3}$	99	104
8	4	$\bar{3}$	34	29
9	4	$\bar{3}$	40	51
0	5	3	543	452
1	5	3	671	-604
1	5	$\bar{3}$	288	-172
2	5	3	604	-576
2	5	$\bar{3}$	149	91
3	5	3	93	72
3	5	$\bar{3}$	640	-637
4	5	3	193	193
4	5	$\bar{3}$	58-	14
5	5	3	35-	21
5	5	$\bar{3}$	283	293
6	5	3	66	63
6	5	$\bar{3}$	206	195
7	5	$\bar{3}$	280	-298
8	5	$\bar{3}$	59	55
9	5	$\bar{3}$	73	-74
0	6	3	477	-417
1	6	3	471	-407
1	6	$\bar{3}$	58-	13
2	6	3	184	160
2	6	$\bar{3}$	161	120
3	6	3	199	190
3	6	$\bar{3}$	114	87
4	6	3	92	-96
4	6	$\bar{3}$	355	368
5	6	3	31-	19
5	6	$\bar{3}$	50-	-14
6	6	3	55	-65
6	6	$\bar{3}$	275	-293
7	6	$\bar{3}$	144	-146
8	6	$\bar{3}$	107	-111
9	6	$\bar{3}$	23-	-33
0	7	3	278	233
1	7	3	58	55
1	7	$\bar{3}$	58-	-33
2	7	3	112	102
2	7	$\bar{3}$	298	267
3	7	3	47-	-21
3	7	$\bar{3}$	76	-63
4	7	3	42-	17
4	7	$\bar{3}$	161	147
5	7	3	66	-97
5	7	$\bar{3}$	50	-47
6	7	$\bar{3}$	172	181
7	7	$\bar{3}$	55	-64
8	7	$\bar{3}$	92	96
0	8	3	94	-79
1	8	3	190	-176
1	8	$\bar{3}$	247	-223
2	8	3	208	198
2	8	$\bar{3}$	78	-77
3	8	3	251	276
3	8	$\bar{3}$	78	-76
4	8	3	34-	-14
4	8	$\bar{3}$	234	272
5	8	3	47	64
5	8	$\bar{3}$	85	93
6	8	$\bar{3}$	99	-103
7	8	$\bar{3}$	43	49
8	8	$\bar{3}$	48	57
0	9	3	140	-122
1	9	3	127	122
1	9	$\bar{3}$	188	-173
2	9	3	270	277
2	9	$\bar{3}$	58-	-8
3	9	3	99	-100
3	9	$\bar{3}$	83	-83
4	9	3	23-	19
4	9	$\bar{3}$	50-	14
5	9	$\bar{3}$	327	-393
6	9	$\bar{3}$	45	-38
7	9	$\bar{3}$	37	-46
0	10	3	55-	-37
1	10	3	38-	40
1	10	$\bar{3}$	44-	-13
2	10	3	144	-156
2	10	$\bar{3}$	50-	26
3	10	3	73	-92
3	10	$\bar{3}$	42-	-47
4	10	$\bar{3}$	74	-81
5	10	$\bar{3}$	29-	-27
6	10	$\bar{3}$	39	32
0	11	3	42-	-22
1	11	3	27-	12
1	11	$\bar{3}$	34-	-13
2	11	3	76	92
2	11	$\bar{3}$	121	127
3	11	$\bar{3}$	55	64
4	11	$\bar{3}$	121	146
5	11	$\bar{3}$	48	-59
0	12	3	24-	47
1	12	$\bar{3}$	21-	-30
2	12	$\bar{3}$	24-	13
3	12	$\bar{3}$	18-	11
0	0	4	393	332
1	0	4	614	-587
1	0	$\bar{4}$	1994	2059
2	0	4	490	-450
2	0	$\bar{4}$	59-	-8
3	0	4	168	173
3	0	$\bar{4}$	566	388
4	0	4	363	-374
4	0	$\bar{4}$	35-	3
5	0	4	38-	10
5	0	$\bar{4}$	821	673
6	0	4	334	-324
6	0	$\bar{4}$	478	450
7	0	$\bar{4}$	187	-174
8	0	$\bar{4}$	248	-236
9	0	$\bar{4}$	75-	44
10	0	$\bar{4}$	20-	5
0	1	4	626	-538
1	1	4	182	-168
1	1	$\bar{4}$	635	-538
2	1	4	412	375
2	1	$\bar{4}$	459	-382
3	1	4	277	271
3	1	$\bar{4}$	47-	25
4	1	4	188	190
4	1	$\bar{4}$	107	-63
5	1	4	37-	-6
5	1	$\bar{4}$	43	49
6	1	4	22-	20
6	1	$\bar{4}$	208	-176
7	1	$\bar{4}$	145	-131
8	1	$\bar{4}$	173	163
9	1	$\bar{4}$	133	125
10	1	$\bar{4}$	90	87
0	2	4	315	-215
1	2	4	58-	0
1	2	$\bar{4}$	419	335
2	2	4	360	-322
2	2	$\bar{4}$	914	-817
3	2	4	317	307
3	2	$\bar{4}$	83	-40
4	2	4	60	-49
4	2	$\bar{4}$	275	-250
5	2	4	241	248
5	2	$\bar{4}$	98	-66
6	2	4	50	-46
6	2	$\bar{4}$	202	-191
7	2	$\bar{4}$	74	-62
8	2	$\bar{4}$	124	-119
9	2	$\bar{4}$	63-	-13
10	2	$\bar{4}$	39-	-8
0	3	4	1276	-1327
1	3	4	599	-505
1	3	$\bar{4}$	57-	-53
2	3	4	90	89
2	3	$\bar{4}$	446	-387
3	3	4	209	-198
3	3	$\bar{4}$	52-	-16
4	3	4	87	-78
4	3	$\bar{4}$	169	154
5	3	4	98	-89
5	3	$\bar{4}$	161	143
6	3	4	18-	11
6	3	$\bar{4}$	358	-370
7	3	$\bar{4}$	415	-418
8	3	$\bar{4}$	62	52
9	3	$\bar{4}$	59-	3
10	3	$\bar{4}$	36-	-23
0	4	4	687	-592
1	4	4	793	754
1	4	$\bar{4}$	381	-313
2	4	4	65-	47
2	4	$\bar{4}$	64-	2
3	4	4	220	206
3	4	$\bar{4}$	310	283
4	4	4	230	-238
4	4	$\bar{4}$	57	53
5	4	4	35	28
5	4	$\bar{4}$	57	51
6	4	4	13	10
6	4	$\bar{4}$	196	-191
7	4	$\bar{4}$	355	366
8	4	$\bar{4}$	190	189
9	4	$\bar{4}$	47-	35
10	4	$\bar{4}$	24-	-3
0	5	4	349	283

Table 4 (*cont.*)

h	k	l	$\lvert 10F_o \rvert$	$10F_c$
1	5	4	293	237
1	5	$\bar{4}$	402	-312
2	5	4	149	136
2	5	$\bar{4}$	66–	-6
3	5	4	93	89
3	5	$\bar{4}$	95	69
4	5	4	45–	24
4	5	$\bar{4}$	58–	48
5	5	4	27–	19
5	5	$\bar{4}$	94	-71
6	5	$\bar{4}$	229	231
7	5	$\bar{4}$	90	94
8	5	$\bar{4}$	27–	24
9	5	$\bar{4}$	38–	0
0	6	4	414	-336
1	6	4	537	487
1	6	$\bar{4}$	346	-276
2	6	4	147	-129
2	6	$\bar{4}$	244	-189
3	6	4	123	122
3	6	$\bar{4}$	121	115
4	6	4	63	-57
4	6	$\bar{4}$	149	-117
5	6	4	90	97
5	6	$\bar{4}$	129	-128
6	6	$\bar{4}$	308	-318
7	6	$\bar{4}$	90	94
8	6	$\bar{4}$	68	53
9	6	$\bar{4}$	30–	-19
0	7	4	403	364
1	7	4	265	232
1	7	$\bar{4}$	237	198
2	7	4	173	-150
2	7	$\bar{4}$	328	278
3	7	4	68	-67
3	7	$\bar{4}$	56–	-6
4	7	4	59	-61
4	7	$\bar{4}$	85	76
5	7	4	11–	69
5	7	$\bar{4}$	163	-183
6	7	$\bar{4}$	213	221
7	7	$\bar{4}$	128	131
8	7	$\bar{4}$	74	-70
9	7	$\bar{4}$	15–	-67
0	8	4	63	-38
1	8	4	94	-76
1	8	$\bar{4}$	438	372
2	8	4	198	-199
2	8	$\bar{4}$	63–	21
3	8	4	40	34
3	8	$\bar{4}$	174	167
4	8	4	80	-106
4	8	$\bar{4}$	71	-58
5	8	$\bar{4}$	168	191
6	8	$\bar{4}$	80	79
7	8	$\bar{4}$	83	-84
8	8	$\bar{4}$	62	-73
0	9	4	45	44
1	9	4	42–	11
1	9	$\bar{4}$	40	-34
2	9	4	78	-73
2	9	$\bar{4}$	66	61
3	9	4	25–	21
3	9	$\bar{4}$	82	-76
4	9	$\bar{4}$	49–	-6
5	9	$\bar{4}$	119	-127
6	9	$\bar{4}$	48	47
7	9	$\bar{4}$	38	33
0	10	4	79	71
1	10	4	60	-47
1	10	$\bar{4}$	253	235
2	10	4	95	-100
2	10	$\bar{4}$	139	-129
3	10	$\bar{4}$	40–	38
4	10	$\bar{4}$	84	-92
5	10	$\bar{4}$	63	63
6	10	$\bar{4}$	74	78
7	10	$\bar{4}$	8–	-50
0	11	4	190	-209
1	11	4	21–	-36
1	11	$\bar{4}$	60	-52
2	11	$\bar{4}$	36–	-10
3	11	$\bar{4}$	31	-40
4	11	$\bar{4}$	39	41
5	11	$\bar{4}$	19–	14
1	12	$\bar{4}$	15–	-13
2	12	$\bar{4}$	40	-57
3	12	$\bar{4}$	15–	-1
0	1	5	75–	95
1	1	5	196	-155
1	1	$\bar{5}$	976	-1070
2	1	5	246	242
2	1	$\bar{5}$	218	-182
3	1	5	132	-123
3	1	$\bar{5}$	405	383
4	1	5	57	-56
4	1	$\bar{5}$	297	273
5	1	5	74	62
5	1	$\bar{5}$	36–	-18
6	1	$\bar{5}$	156	-168
7	1	$\bar{5}$	119	108
8	1	$\bar{5}$	214	215
9	1	$\bar{5}$	58–	12
10	1	$\bar{5}$	139	-125
0	2	5	476	426
1	2	5	58–	-31
1	2	$\bar{5}$	97	66
2	2	5	65–	27
2	2	$\bar{5}$	1023	1156
3	2	5	142	-143
3	2	$\bar{5}$	498	445
4	2	5	152	139
4	2	$\bar{5}$	211	-210
5	2	5	213	223
5	2	$\bar{5}$	41–	-73
6	2	$\bar{5}$	64	-51
7	2	$\bar{5}$	239	240
8	2	$\bar{5}$	133	-127
9	2	$\bar{5}$	249	-241
10	2	$\bar{5}$	47–	-38
0	3	5	355	299
1	3	5	239	-213
1	3	$\bar{5}$	585	484
2	3	5	113	94
2	3	$\bar{5}$	479	382
3	3	5	171	-161
3	3	$\bar{5}$	362	-313
4	3	5	48	59
4	3	$\bar{5}$	158	105
5	3	5	173	-197
5	3	$\bar{5}$	45–	5
6	3	$\bar{5}$	39–	29
7	3	$\bar{5}$	35–	11
8	3	$\bar{5}$	144	-132
9	3	$\bar{5}$	64–	64
10	3	$\bar{5}$	43–	16
0	4	5	455	378
1	4	5	157	137
1	4	$\bar{5}$	211	-156
2	4	5	169	143
2	4	$\bar{5}$	234	186
3	4	5	47–	-39
3	4	$\bar{5}$	161	149
4	4	5	76	74
4	4	$\bar{5}$	129	-165
5	4	5	80	95
5	4	$\bar{5}$	47–	12
6	4	$\bar{5}$	42–	17
7	4	$\bar{5}$	204	208
8	4	$\bar{5}$	29–	0
9	4	$\bar{5}$	51–	-39
10	4	$\bar{5}$	32–	16
0	5	5	128	101
1	5	5	182	-176
1	5	$\bar{5}$	585	495
2	5	5	198	171
2	5	$\bar{5}$	166	126
3	5	5	44–	22
3	5	$\bar{5}$	720	-729
4	5	5	267	301
4	5	$\bar{5}$	180	-168
5	5	5	14–	-40
5	5	$\bar{5}$	152	-139
6	5	$\bar{5}$	43–	11
7	5	$\bar{5}$	63	-65
8	5	$\bar{5}$	264	-281
9	5	$\bar{5}$	42–	21
10	5	$\bar{5}$	23–	40
0	6	5	464	-429
1	6	5	249	-225
1	6	$\bar{5}$	277	206
2	6	5	139	-126
2	6	$\bar{5}$	642	-581
3	6	5	74	-84
3	6	$\bar{5}$	56–	-22
4	6	5	95	-110
4	6	$\bar{5}$	59–	7
5	6	$\bar{5}$	50–	18
6	6	$\bar{5}$	54	-76
7	6	$\bar{5}$	95	-96
8	6	$\bar{5}$	26–	-12
9	6	$\bar{5}$	113	108
0	7	5	82	66
1	7	5	180	-166
1	7	$\bar{5}$	55–	-38
2	7	5	49–	3
2	7	$\bar{5}$	218	178
3	7	5	121	-131
3	7	$\bar{5}$	62	48
4	7	5	21–	22
4	7	$\bar{5}$	185	174
5	7	$\bar{5}$	147	150
6	7	$\bar{5}$	93	111
7	7	$\bar{5}$	83	88
8	7	$\bar{5}$	22–	-2
9	7	$\bar{5}$	20–	23
0	8	5	63–	-21
1	8	5	125	119
1	8	$\bar{5}$	51–	-4
2	8	5	123	121
2	8	$\bar{5}$	507	-488
3	8	5	58	75
3	8	$\bar{5}$	52–	1
4	8	$\bar{5}$	55–	21
5	8	$\bar{5}$	53	54
6	8	$\bar{5}$	36–	-10
7	8	$\bar{5}$	29–	-22
8	8	$\bar{5}$	18–	-7
0	9	5	52	49
1	9	5	36–	-9
1	9	$\bar{5}$	477	-458
2	9	5	84	87
2	9	$\bar{5}$	107	-86
3	9	5	10–	-13
3	9	$\bar{5}$	123	110
4	9	$\bar{5}$	47–	-47
5	9	$\bar{5}$	54	-61
6	9	$\bar{5}$	76	-81
7	9	$\bar{5}$	22–	-11
8	9	$\bar{5}$	10–	72
0	10	5	42–	-4
1	10	5	67	-75
1	10	$\bar{5}$	128	128
2	10	5	15–	18
2	10	$\bar{5}$	137	124
3	10	$\bar{5}$	135	149
4	10	$\bar{5}$	39–	-5
5	10	$\bar{5}$	45	49
6	10	$\bar{5}$	60	-66
7	10	$\bar{5}$	72–	12
0	11	5	90	89
1	11	$\bar{5}$	93	-95
2	11	$\bar{5}$	54	67
3	11	$\bar{5}$	87	91
4	11	$\bar{5}$	27–	32
5	11	$\bar{5}$	18–	21
0	0	6	148	110
1	0	6	423	-428
1	0	$\bar{6}$	174	222
2	0	6	163	-151
2	0	$\bar{6}$	663	670
3	0	6	219	204
3	0	$\bar{6}$	888	-1016
4	0	6	119	99
4	0	$\bar{6}$	158	-155
5	0	$\bar{6}$	429	388
6	0	$\bar{6}$	438	396
7	0	$\bar{6}$	18–	7
8	0	$\bar{6}$	189	162
9	0	$\bar{6}$	117	-76
10	0	$\bar{6}$	289	224
11	0	$\bar{6}$	49–	25
0	1	6	428	360
1	1	6	286	252
1	1	$\bar{6}$	293	-254
2	1	6	259	235
2	1	$\bar{6}$	686	-715
3	1	6	94	86
3	1	$\bar{6}$	747	-786
4	1	6	177	-174
4	1	$\bar{6}$	246	219
5	1	6	14–	-86
5	1	$\bar{6}$	299	284
6	1	$\bar{6}$	113	89
7	1	$\bar{6}$	83	57
8	1	$\bar{6}$	35–	34
9	1	$\bar{6}$	187	170
10	1	$\bar{6}$	103	90
11	1	$\bar{6}$	24–	-22
0	2	6	142	136
1	2	6	57–	8
1	2	$\bar{6}$	169	-129
2	2	6	277	267
2	2	$\bar{6}$	337	247
3	2	6	124	117
3	2	$\bar{6}$	604	-575
4	2	6	129	137
4	2	$\bar{6}$	251	-215
5	2	6	10–	37
5	2	$\bar{6}$	384	-348
6	2	$\bar{6}$	37–	16
7	2	$\bar{6}$	222	-229
8	2	$\bar{6}$	24–	-10
9	2	$\bar{6}$	157	-158
10	2	$\bar{6}$	80	64
11	2	$\bar{6}$	25–	-22
0	3	6	344	-316
1	3	6	197	-167
1	3	$\bar{6}$	353	280
2	3	6	169	164
2	3	$\bar{6}$	388	-349
3	3	6	137	128
3	3	$\bar{6}$	368	306
4	3	6	220	-235
4	3	$\bar{6}$	704	713
5	3	$\bar{6}$	315	297
6	3	$\bar{6}$	54	-33
7	3	$\bar{6}$	35–	-4
8	3	$\bar{6}$	90	-90
9	3	$\bar{6}$	68	59
10	3	$\bar{6}$	49–	15
11	3	$\bar{6}$	13–	-53
0	4	6	76	68
1	4	6	249	-225
1	4	$\bar{6}$	265	-204
2	4	6	56–	2
2	4	$\bar{6}$	104	-80
3	4	6	376	-401
3	4	$\bar{6}$	542	503
4	4	6	137	-152
4	4	$\bar{6}$	860	906
5	4	$\bar{6}$	103	-93
6	4	$\bar{6}$	156	142
7	4	$\bar{6}$	87	-73
8	4	$\bar{6}$	272	207
9	4	$\bar{6}$	54–	5
10	4	$\bar{6}$	71	-59
0	5	6	73–	26
1	5	6	188	189
1	5	$\bar{6}$	69	-56
2	5	6	52–	-44
2	5	$\bar{6}$	66–	12
3	5	6	34–	20
3	5	$\bar{6}$	93	-71
4	5	6	54	68
4	5	$\bar{6}$	196	-159
5	5	$\bar{6}$	166	-157
6	5	$\bar{6}$	43–	-45
7	5	$\bar{6}$	37–	-14
8	5	$\bar{6}$	28–	8
9	5	$\bar{6}$	45–	7
10	5	$\bar{6}$	45	44
0	6	6	138	117
1	6	6	49–	29
1	6	$\bar{6}$	261	-220
2	6	6	197	191
2	6	$\bar{6}$	90	-77
3	6	6	127	-139
3	6	$\bar{6}$	139	123
4	6	$\bar{6}$	289	251
5	6	$\bar{6}$	315	-323
6	6	$\bar{6}$	42–	10
7	6	$\bar{6}$	108	-134
8	6	$\bar{6}$	48	45
9	6	$\bar{6}$	38–	-33
10	6	$\bar{6}$	17–	-30
0	7	6	172	-160
1	7	6	74	35
1	7	$\bar{6}$	102	-71
2	7	6	184	-173
2	7	$\bar{6}$	344	304
3	7	6	67	-84
3	7	$\bar{6}$	441	401
4	7	$\bar{6}$	211	-181
5	7	$\bar{6}$	206	-207
6	7	$\bar{6}$	40–	23
7	7	$\bar{6}$	34–	15
8	7	$\bar{6}$	23–	23
9	7	$\bar{6}$	92	-103
0	8	6	145	128
1	8	6	76	-54
1	8	$\bar{6}$	55	-48
2	8	6	30–	23
2	8	$\bar{6}$	140	105
3	8	$\bar{6}$	333	-308
4	8	$\bar{6}$	98	-98
5	8	$\bar{6}$	43–	-22
6	8	$\bar{6}$	90	97
7	8	$\bar{6}$	29–	-1
8	8	$\bar{6}$	52	64
9	8	$\bar{6}$	10–	-2
0	9	6	45–	-10
1	9	6	37	68
1	9	$\bar{6}$	83	-77
2	9	6	16–	45
2	9	$\bar{6}$	47	43
3	9	$\bar{6}$	28	20
4	9	$\bar{6}$	53	-47
5	9	$\bar{6}$	43	-41
6	9	$\bar{6}$	37	35
7	9	$\bar{6}$	22–	-9
8	9	$\bar{6}$	11–	7
0	10	6	32–	27
1	10	6	13–	-56
1	10	$\bar{6}$	30–	-38
2	10	$\bar{6}$	198	202
3	10	$\bar{6}$	142	-145
4	10	$\bar{6}$	85	-82
5	10	$\bar{6}$	42	37
6	10	$\bar{6}$	63	67
7	10	$\bar{6}$	12–	-34
1	11	$\bar{6}$	17–	-50
2	11	$\bar{6}$	25–	-90
3	11	$\bar{6}$	102	-123
4	11	$\bar{6}$	98	121
5	11	$\bar{6}$	45	53
0	1	7	209	-173
1	1	7	574	595
1	1	$\bar{7}$	384	352
2	1	7	121	-109
2	1	$\bar{7}$	275	-291
3	1	7	135	-123
3	1	$\bar{7}$	352	351
4	1	7	74	-96
4	1	$\bar{7}$	214	208
5	1	$\bar{7}$	164	139
6	1	$\bar{7}$	708	-777
7	1	$\bar{7}$	113	100
8	1	$\bar{7}$	112	-125
9	1	$\bar{7}$	100	92
10	1	$\bar{7}$	78	-81
11	1	$\bar{7}$	29–	-13
0	2	7	201	197
1	2	7	163	-136
1	2	$\bar{7}$	153	119
2	2	7	455	-459
2	2	$\bar{7}$	76–	39
3	2	7	34–	-35
3	2	$\bar{7}$	367	364
4	2	7	76	94
4	2	$\bar{7}$	142	-135
5	2	$\bar{7}$	658	-754
6	2	$\bar{7}$	39–	29
7	2	$\bar{7}$	218	211
8	2	$\bar{7}$	25–	34
9	2	$\bar{7}$	71–	-9
10	2	$\bar{7}$	55–	-48
11	2	$\bar{7}$	32–	29
0	3	7	413	-382
1	3	7	123	-111
1	3	$\bar{7}$	204	182
2	3	7	128	-131
2	3	$\bar{7}$	403	-347
3	3	7	153	159
3	3	$\bar{7}$	246	198
4	3	7	13–	-47
4	3	$\bar{7}$	461	-406
5	3	$\bar{7}$	492	482
6	3	$\bar{7}$	135	112
7	3	$\bar{7}$	288	287
8	3	$\bar{7}$	27–	-31
9	3	$\bar{7}$	84	93
10	3	$\bar{7}$	52–	-38
11	3	$\bar{7}$	26–	51
0	4	7	216	190
1	4	7	49–	48
1	4	$\bar{7}$	80	-71
2	4	7	164	-148
2	4	$\bar{7}$	268	-220
3	4	7	52	-62
3	4	$\bar{7}$	56–	-20
4	4	$\bar{7}$	129	-106
5	4	$\bar{7}$	402	-377
6	4	$\bar{7}$	42–	3
7	4	$\bar{7}$	104	102
8	4	$\bar{7}$	29–	40
9	4	$\bar{7}$	55–	15
10	4	$\bar{7}$	39–	26
0	5	7	142	-126
1	5	7	161	-136
1	5	$\bar{7}$	348	308
2	5	7	41–	8
2	5	$\bar{7}$	241	-188
3	5	7	275	321
3	5	$\bar{7}$	135	107
4	5	$\bar{7}$	753	-778
5	5	$\bar{7}$	211	179
6	5	$\bar{7}$	104	104
7	5	$\bar{7}$	60	52
8	5	$\bar{7}$	153	-165
9	5	$\bar{7}$	47–	12
10	5	$\bar{7}$	31–	-42
0	6	7	196	-175
1	6	7	73	51
1	6	$\bar{7}$	152	135
2	6	7	251	267
2	6	$\bar{7}$	189	140
3	6	7	14–	4
3	6	$\bar{7}$	107	83
4	6	$\bar{7}$	360	318
5	6	$\bar{7}$	412	409
6	6	$\bar{7}$	55	26
7	6	$\bar{7}$	113	-109
8	6	$\bar{7}$	58	-46
9	6	$\bar{7}$	85	-94
10	6	$\bar{7}$	22–	2
0	7	7	148	-141
1	7	7	34–	15
1	7	$\bar{7}$	135	124
2	7	7	139	-178
2	7	$\bar{7}$	169	-141
3	7	$\bar{7}$	284	264
4	7	$\bar{7}$	112	-96
5	7	$\bar{7}$	206	206
6	7	$\bar{7}$	39–	-23
7	7	$\bar{7}$	129	142
8	7	$\bar{7}$	33	-33
9	7	$\bar{7}$	84	92
0	8	7	53	-48
1	8	7	26–	22
1	8	$\bar{7}$	90	-79
2	8	$\bar{7}$	145	-138
3	8	$\bar{7}$	261	-246
4	8	$\bar{7}$	58	-54
5	8	$\bar{7}$	158	159
6	8	$\bar{7}$	34–	-28
7	8	$\bar{7}$	78	-83
8	8	$\bar{7}$	53	53
9	8	$\bar{7}$	14–	8
0	9	7	34–	-6
1	9	$\bar{7}$	127	126
2	9	$\bar{7}$	95	-89
3	9	$\bar{7}$	133	127
4	9	$\bar{7}$	35	27
5	9	$\bar{7}$	50	-37
6	9	$\bar{7}$	307	-307
7	9	$\bar{7}$	21–	-4
8	9	$\bar{7}$	108	-109
0	10	7	11–	-14
1	10	$\bar{7}$	50	75
2	10	$\bar{7}$	45	46
3	10	$\bar{7}$	76	75
4	10	$\bar{7}$	31–	7
5	10	$\bar{7}$	64	-65
6	10	$\bar{7}$	31	32
7	10	$\bar{7}$	10–	0
3	11	$\bar{7}$	15–	70
4	11	$\bar{7}$	17–	24
0	0	8	495	-495
1	0	8	543	548
1	0	$\bar{8}$	56–	71
2	0	8	55	47
2	0	$\bar{8}$	206	181
3	0	8	145	-159
3	0	$\bar{8}$	128	-136
4	0	$\bar{8}$	139	131
5	0	$\bar{8}$	48–	-16
6	0	$\bar{8}$	715	717
7	0	$\bar{8}$	403	354
8	0	$\bar{8}$	37	-39
9	0	$\bar{8}$	57–	-7
10	0	$\bar{8}$	35–	32
11	0	$\bar{8}$	50–	47
0	1	8	293	-273
1	1	8	211	194
1	1	$\bar{8}$	206	205
2	1	8	138	122
2	1	$\bar{8}$	320	-304
3	1	8	22–	-9
3	1	$\bar{8}$	198	-227
4	1	$\bar{8}$	193	-199
5	1	$\bar{8}$	48–	3
6	1	$\bar{8}$	133	-124
7	1	$\bar{8}$	378	-368
8	1	$\bar{8}$	124	-117
9	1	$\bar{8}$	61	53
10	1	$\bar{8}$	50–	43
11	1	$\bar{8}$	55	58
0	2	8	111	77
1	2	8	108	-84
1	2	$\bar{8}$	197	194
2	2	8	209	212
2	2	$\bar{8}$	304	290
3	2	8	20–	5
3	2	$\bar{8}$	182	-164
4	2	$\bar{8}$	43	13
5	2	$\bar{8}$	135	-136
6	2	$\bar{8}$	42–	12
7	2	$\bar{8}$	172	-173
8	2	$\bar{8}$	166	-142
9	2	$\bar{8}$	152	-112
10	2	$\bar{8}$	55–	-54
11	2	$\bar{8}$	35–	-27
0	3	8	393	-382
1	3	8	184	167
1	3	$\bar{8}$	488	445
2	3	8	100	96
2	3	$\bar{8}$	65–	38
3	3	8	16–	24
3	3	$\bar{8}$	267	236
4	3	$\bar{8}$	143	126
5	3	$\bar{8}$	413	410
6	3	$\bar{8}$	278	236
7	3	$\bar{8}$	270	-261
8	3	$\bar{8}$	166	-159
9	3	$\bar{8}$	68–	22
10	3	$\bar{8}$	52–	-24
11	3	$\bar{8}$	31–	-9
0	4	8	342	319
1	4	8	174	160
1	4	$\bar{8}$	441	-415
2	4	8	32–	-32
2	4	$\bar{8}$	129	-122
3	4	$\bar{8}$	132	-116
4	4	$\bar{8}$	114	118
5	4	$\bar{8}$	50–	1
6	4	$\bar{8}$	293	-260
7	4	$\bar{8}$	114	-70
8	4	$\bar{8}$	293	299
9	4	$\bar{8}$	97	108
10	4	$\bar{8}$	73	75
11	4	$\bar{8}$	18–	26
0	5	8	145	102
1	5	8	36–	17
1	5	$\bar{8}$	123	98
2	5	8	27–	-11
2	5	$\bar{8}$	267	-236
3	5	$\bar{8}$	180	-151
4	5	$\bar{8}$	104	-88
5	5	$\bar{8}$	124	-99
6	5	$\bar{8}$	114	-110
7	5	$\bar{8}$	100	97
8	5	$\bar{8}$	28–	13
9	5	$\bar{8}$	46–	30
10	5	$\bar{8}$	32–	-6
0	6	8	244	226
1	6	8	104	93
1	6	$\bar{8}$	175	-158
2	6	8	18–	48
2	6	$\bar{8}$	66	37
3	6	$\bar{8}$	83	-61
4	6	$\bar{8}$	73	71
5	6	$\bar{8}$	47–	14
6	6	$\bar{8}$	190	-168
7	6	$\bar{8}$	188	-183
8	6	$\bar{8}$	67	61
9	6	$\bar{8}$	39–	-24
10	6	$\bar{8}$	23–	-24
0	7	8	246	236
1	7	8	135	-156
1	7	$\bar{8}$	138	-122
2	7	$\bar{8}$	111	87
3	7	$\bar{8}$	67	61
4	7	$\bar{8}$	50–	33
5	7	$\bar{8}$	107	-101
6	7	$\bar{8}$	99	-87
7	7	$\bar{8}$	272	294
8	7	$\bar{8}$	62	59
9	7	$\bar{8}$	23–	-4
0	8	8	79	-78
1	8	$\bar{8}$	32–	-5
2	8	$\bar{8}$	71	64
3	8	$\bar{8}$	40	-26
4	8	$\bar{8}$	80	81
5	8	$\bar{8}$	53	-37
6	8	$\bar{8}$	180	183
7	8	$\bar{8}$	40	35
8	8	$\bar{8}$	43	-38
9	8	$\bar{8}$	13–	-26
0	9	8	11–	56
1	9	$\bar{8}$	23–	20
2	9	$\bar{8}$	33–	-5
3	9	$\bar{8}$	37	37
4	9	$\bar{8}$	35–	14
5	9	$\bar{8}$	29–	5
6	9	$\bar{8}$	95	-96
7	9	$\bar{8}$	45	34
8	9	$\bar{8}$	9–	-20
2	10	$\bar{8}$	19–	35
3	10	$\bar{8}$	21–	-45
4	10	$\bar{8}$	24–	0
5	10	$\bar{8}$	57	-65
6	10	$\bar{8}$	125	144
0	1	9	175	152
1	1	9	90	70
1	1	$\bar{9}$	168	-138
2	1	9	26–	29
2	1	$\bar{9}$	402	-412
3	1	$\bar{9}$	85	-85
4	1	$\bar{9}$	142	175
5	1	$\bar{9}$	68	-53
6	1	$\bar{9}$	93	71
7	1	$\bar{9}$	304	-298
8	1	$\bar{9}$	263	261
9	1	$\bar{9}$	103	82
10	1	$\bar{9}$	48–	23
11	1	$\bar{9}$	74	-74
0	2	9	154	122
1	2	9	34–	30
1	2	$\bar{9}$	469	-484
2	2	9	23–	2
2	2	$\bar{9}$	175	149
3	2	$\bar{9}$	377	398
4	2	$\bar{9}$	129	130
5	2	$\bar{9}$	133	99
6	2	$\bar{9}$	42–	1
7	2	$\bar{9}$	209	205
8	2	$\bar{9}$	284	280
9	2	$\bar{9}$	145	-131
10	2	$\bar{9}$	92	-117
11	2	$\bar{9}$	37–	-26
0	3	9	171	-146
1	3	9	31–	-15
1	3	$\bar{9}$	60	57
2	3	9	64	-81
2	3	$\bar{9}$	317	285
3	3	$\bar{9}$	83	-81
4	3	$\bar{9}$	58–	41
5	3	$\bar{9}$	104	-86
6	3	$\bar{9}$	133	123

Table 4 (*cont.*)

h	k	l	$\lvert 10F_o\rvert$	$10F_c$
7	3	$\bar{9}$	53	−20
8	3	$\bar{9}$	69	47
9	3	$\bar{9}$	133	−121
10	3	$\bar{9}$	95	98
11	3	$\bar{9}$	31−	−11
0	4	9	50−	5
1	4	9	28−	20
1	4	$\bar{9}$	234	−215
2	4	$\bar{9}$	69	38
3	4	$\bar{9}$	52−	26
4	4	$\bar{9}$	57−	−26
5	4	$\bar{9}$	130	−138
6	4	$\bar{9}$	140	−126
7	4	$\bar{9}$	37−	−27
8	4	$\bar{9}$	107	103
9	4	$\bar{9}$	52−	−56
10	4	$\bar{9}$	40−	−20
11	4	$\bar{9}$	19−	25
0	5	9	147	−137
1	5	9	48	40
1	5	$\bar{9}$	184	179
2	5	$\bar{9}$	522	501
3	5	$\bar{9}$	62	−45
4	5	$\bar{9}$	55−	24
5	5	$\bar{9}$	90	−82
6	5	$\bar{9}$	85	67
7	5	$\bar{9}$	36−	−2
8	5	$\bar{9}$	127	−128
9	5	$\bar{9}$	249	−279
10	5	$\bar{9}$	31−	26
0	6	9	128	−135
1	6	9	15−	27
1	6	$\bar{9}$	291	274
2	6	$\bar{9}$	48−	37
3	6	$\bar{9}$	219	−202
4	6	$\bar{9}$	128	123
5	6	$\bar{9}$	43−	−12
6	6	$\bar{9}$	125	118
7	6	$\bar{9}$	54	−51
8	6	$\bar{9}$	66	−68
9	6	$\bar{9}$	57	57
10	6	$\bar{9}$	55	56
0	7	9	55	−65
1	7	$\bar{9}$	59	−73
2	7	$\bar{9}$	66	−54
3	7	$\bar{9}$	112	−112
4	7	$\bar{9}$	45−	1
5	7	$\bar{9}$	64	−60
6	7	$\bar{9}$	112	107
7	7	$\bar{9}$	31	35
8	7	$\bar{9}$	132	141
9	7	$\bar{9}$	21−	5
1	8	$\bar{9}$	87	95
2	8	$\bar{9}$	48	−66
3	8	$\bar{9}$	218	−243
4	8	$\bar{9}$	37−	26
5	8	$\bar{9}$	105	−117
6	8	$\bar{9}$	27−	3
7	8	$\bar{9}$	97	−105
8	8	$\bar{9}$	98	−104
2	9	$\bar{9}$	135	−165
3	9	$\bar{9}$	23	20
4	9	$\bar{9}$	66	69
5	9	$\bar{9}$	43	−53
6	9	$\bar{9}$	20−	18
7	9	$\bar{9}$	130	−142
5	10	$\bar{9}$	10−	43
0	0	10	145	−139
1	0	10	22−	−28
1	0	$\bar{10}$	43−	−17
2	0	$\bar{10}$	286	278
3	0	$\bar{10}$	211	197
4	0	$\bar{10}$	562	726
5	0	$\bar{10}$	49−	−31
6	0	$\bar{10}$	107	−100
7	0	$\bar{10}$	118	142
8	0	$\bar{10}$	38	−37
9	0	$\bar{10}$	24−	6
10	0	$\bar{10}$	25−	6
11	0	$\bar{10}$	148	224
0	1	10	45−	24
1	1	10	45	−48
1	1	$\bar{10}$	58	−34
2	1	$\bar{10}$	80	−86
3	1	$\bar{10}$	416	−437
4	1	$\bar{10}$	275	−269
5	1	$\bar{10}$	48−	−29
6	1	$\bar{10}$	129	−115
7	1	$\bar{10}$	40	−43
8	1	$\bar{10}$	105	−109
9	1	$\bar{10}$	57−	−37
10	1	$\bar{10}$	100	98
11	1	$\bar{10}$	31−	18
0	2	10	42−	41
1	2	10	109	115
1	2	$\bar{10}$	154	149
2	2	$\bar{10}$	58	58
3	2	$\bar{10}$	201	181
4	2	$\bar{10}$	178	−160
5	2	$\bar{10}$	156	152
6	2	$\bar{10}$	168	−151
7	2	$\bar{10}$	58	57
8	2	$\bar{10}$	129	−138
9	2	$\bar{10}$	65−	−18
10	2	$\bar{10}$	148	−152
11	2	$\bar{10}$	34−	23
0	3	10	39−	15
1	3	10	15−	5
1	3	$\bar{10}$	193	179
2	3	$\bar{10}$	135	110
3	3	$\bar{10}$	402	−405
4	3	$\bar{10}$	78	−74
5	3	$\bar{10}$	333	336
6	3	$\bar{10}$	111	107
7	3	$\bar{10}$	130	122
8	3	$\bar{10}$	28−	11
9	3	$\bar{10}$	62−	47
10	3	$\bar{10}$	166	162
11	3	$\bar{10}$	30−	11
0	4	10	203	−213
1	4	$\bar{10}$	36−	−36
2	4	$\bar{10}$	373	−374
3	4	$\bar{10}$	95	−84
4	4	$\bar{10}$	202	189
5	4	$\bar{10}$	248	233
6	4	$\bar{10}$	60	−51
7	4	$\bar{10}$	153	150
8	4	$\bar{10}$	63	−53
9	4	$\bar{10}$	218	247
10	4	$\bar{10}$	37−	−22
0	5	10	27−	35
1	5	$\bar{10}$	31−	−1
2	5	$\bar{10}$	44−	−38
3	5	$\bar{10}$	76	70
4	5	$\bar{10}$	48−	1
5	5	$\bar{10}$	103	−88
6	5	$\bar{10}$	127	−115
7	5	$\bar{10}$	76	−68
8	5	$\bar{10}$	42	−43
9	5	$\bar{10}$	74	−73
10	5	$\bar{10}$	33	−38
1	6	$\bar{10}$	121	141
2	6	$\bar{10}$	175	−166
3	6	$\bar{10}$	37−	27
4	6	$\bar{10}$	161	153
5	6	$\bar{10}$	116	117
6	6	$\bar{10}$	154	−138
7	6	$\bar{10}$	60	52
8	6	$\bar{10}$	93	−105
9	6	$\bar{10}$	88	104
10	6	$\bar{10}$	18−	−56
1	7	$\bar{10}$	16−	16
2	7	$\bar{10}$	40	−37
3	7	$\bar{10}$	264	298
4	7	$\bar{10}$	152	145
5	7	$\bar{10}$	54	−42
6	7	$\bar{10}$	29−	18
7	7	$\bar{10}$	25−	−1
8	7	$\bar{10}$	62	64
9	7	$\bar{10}$	18−	3
2	8	$\bar{10}$	31	35
3	8	$\bar{10}$	54	65
4	8	$\bar{10}$	178	−199
5	8	$\bar{10}$	26−	−15
6	8	$\bar{10}$	57	−70
7	8	$\bar{10}$	47	52
8	8	$\bar{10}$	34	−49
4	9	$\bar{10}$	16−	−26
5	9	$\bar{10}$	43	−55
6	9	$\bar{10}$	13−	−16
0	1	11	121	171
1	1	11	148	−139
2	1	$\bar{11}$	232	218
3	1	$\bar{11}$	202	−201
4	1	$\bar{11}$	312	330
5	1	$\bar{11}$	219	218
6	1	$\bar{11}$	40−	−31
7	1	$\bar{11}$	201	−208
8	1	$\bar{11}$	35	33
9	1	$\bar{11}$	52−	−58
10	1	$\bar{11}$	42−	32
11	1	$\bar{11}$	29−	−83
0	2	11	21−	13
1	2	$\bar{11}$	90	86
2	2	$\bar{11}$	68	51
3	2	$\bar{11}$	140	135
4	2	$\bar{11}$	288	291
5	2	$\bar{11}$	225	−224
6	2	$\bar{11}$	306	−308
7	2	$\bar{11}$	63	52
8	2	$\bar{11}$	19−	1
9	2	$\bar{11}$	60	46
10	2	$\bar{11}$	47−	−41
11	2	$\bar{11}$	31−	−26
0	3	11	7−	19
1	3	$\bar{11}$	153	−153
2	3	$\bar{11}$	78	68
3	3	$\bar{11}$	166	−160
4	3	$\bar{11}$	53	−46
5	3	$\bar{11}$	267	−247
6	3	$\bar{11}$	184	161
7	3	$\bar{11}$	34−	−10
8	3	$\bar{11}$	107	105
9	3	$\bar{11}$	56−	11
10	3	$\bar{11}$	44−	41
11	3	$\bar{11}$	25−	41
1	4	$\bar{11}$	23−	1
2	4	$\bar{11}$	62	−62
3	4	$\bar{11}$	81	−35
4	4	$\bar{11}$	45	48
5	4	$\bar{11}$	113	−112
6	4	$\bar{11}$	211	−190
7	4	$\bar{11}$	48	20
8	4	$\bar{11}$	39	−37
9	4	$\bar{11}$	44−	−5
10	4	$\bar{11}$	40	−42
1	5	$\bar{11}$	15−	−11
2	5	$\bar{11}$	121	119
3	5	$\bar{11}$	32−	13
4	5	$\bar{11}$	68	−67
5	5	$\bar{11}$	372	−356
6	5	$\bar{11}$	193	175
7	5	$\bar{11}$	29−	9
8	5	$\bar{11}$	22−	21
9	5	$\bar{11}$	35−	−42
10	5	$\bar{11}$	23−	−34
2	6	$\bar{11}$	23−	−6
3	6	$\bar{11}$	27−	−4
4	6	$\bar{11}$	74	−67
5	6	$\bar{11}$	261	255
6	6	$\bar{11}$	180	175
7	6	$\bar{11}$	74	76
8	6	$\bar{11}$	31	38
9	6	$\bar{11}$	27−	25
3	7	$\bar{11}$	66	−75
4	7	$\bar{11}$	80	83
5	7	$\bar{11}$	59	−66
6	7	$\bar{11}$	73	70
7	7	$\bar{11}$	23	−24
8	7	$\bar{11}$	14−	22
5	8	$\bar{11}$	45	55
6	8	$\bar{11}$	15−	11
7	8	$\bar{11}$	12−	−47
2	0	$\bar{12}$	125	−125
3	0	$\bar{12}$	153	−140
4	0	$\bar{12}$	149	−151
5	0	$\bar{12}$	161	−149
6	0	$\bar{12}$	36−	18
7	0	$\bar{12}$	341	340
8	0	$\bar{12}$	90	78
9	0	$\bar{12}$	28−	9
10	0	$\bar{12}$	48−	44
11	0	$\bar{12}$	36−	−1
2	1	$\bar{12}$	43	37
3	1	$\bar{12}$	33−	−28
4	1	$\bar{12}$	92	−85
5	1	$\bar{12}$	79	−69
6	1	$\bar{12}$	128	119
7	1	$\bar{12}$	113	−113
8	1	$\bar{12}$	203	−206
9	1	$\bar{12}$	88	−87
10	1	$\bar{12}$	37−	−51
11	1	$\bar{12}$	36	−16
2	2	$\bar{12}$	87	85
3	2	$\bar{12}$	172	180
4	2	$\bar{12}$	39−	25
5	2	$\bar{12}$	42	42
6	2	$\bar{12}$	34−	0
7	2	$\bar{12}$	85	71
8	2	$\bar{12}$	31	−23
9	2	$\bar{12}$	51−	−60
10	2	$\bar{12}$	41−	−22
2	3	$\bar{12}$	24−	14
3	3	$\bar{12}$	50	40
4	3	$\bar{12}$	108	107
5	3	$\bar{12}$	111	101
6	3	$\bar{12}$	310	347
7	3	$\bar{12}$	84	67
8	3	$\bar{12}$	71	−57
9	3	$\bar{12}$	48−	12
10	3	$\bar{12}$	37−	14
3	4	$\bar{12}$	26−	6
4	4	$\bar{12}$	121	−129
5	4	$\bar{12}$	139	128
6	4	$\bar{12}$	156	−96
7	4	$\bar{12}$	272	−263
8	4	$\bar{12}$	28	−18
9	4	$\bar{12}$	49	47
10	4	$\bar{12}$	26	46
3	5	$\bar{12}$	67	−81
4	5	$\bar{12}$	50	−54
5	5	$\bar{12}$	87	−94
6	5	$\bar{12}$	93	−83
7	5	$\bar{12}$	68	−69
8	5	$\bar{12}$	18	9
9	5	$\bar{12}$	28−	−31
4	6	$\bar{12}$	21−	−16
5	6	$\bar{12}$	106	127
6	6	$\bar{12}$	21−	13
7	6	$\bar{12}$	84	−89
8	6	$\bar{12}$	14−	−5
5	7	$\bar{12}$	13−	−16
6	7	$\bar{12}$	83	−143
7	7	$\bar{12}$	12−	0
3	1	$\bar{13}$	19−	−15
4	1	$\bar{13}$	71	−74
5	1	$\bar{13}$	48	46
6	1	$\bar{13}$	59	−41
7	1	$\bar{13}$	55	−46
8	1	$\bar{13}$	173	−164
9	1	$\bar{13}$	180	183
10	1	$\bar{13}$	28−	−7
3	2	$\bar{13}$	16−	56
4	2	$\bar{13}$	26−	−1
5	2	$\bar{13}$	64	57
6	2	$\bar{13}$	26−	16
7	2	$\bar{13}$	33	−16
8	2	$\bar{13}$	225	231
9	2	$\bar{13}$	116	130
10	2	$\bar{13}$	30−	−41
4	3	$\bar{13}$	92	−107
5	3	$\bar{13}$	80	79
6	3	$\bar{13}$	95	−83
7	3	$\bar{13}$	129	118
8	3	$\bar{13}$	28	−28
9	3	$\bar{13}$	37−	9
10	3	$\bar{13}$	24−	−60
4	4	$\bar{13}$	18−	−29
5	4	$\bar{13}$	21−	−10
6	4	$\bar{13}$	118	−78
7	4	$\bar{13}$	105	−98
8	4	$\bar{13}$	23	19
9	4	$\bar{13}$	26−	12
5	5	$\bar{13}$	87	155
6	5	$\bar{13}$	48	−47
7	5	$\bar{13}$	143	168
8	5	$\bar{13}$	26	22
5	0	$\bar{14}$	209	−285
6	0	$\bar{14}$	29	22
7	0	$\bar{14}$	206	−207
8	0	$\bar{14}$	80	83
9	0	$\bar{14}$	133	−134
5	1	$\bar{14}$	15−	−17
6	1	$\bar{14}$	71	−72
7	1	$\bar{14}$	78	−84
8	1	$\bar{14}$	69	−74
9	1	$\bar{14}$	103	−116
5	2	$\bar{14}$	13−	−34
6	2	$\bar{14}$	140	193
7	2	$\bar{14}$	47	−50
8	2	$\bar{14}$	98	110
9	2	$\bar{14}$	25−	−58
6	3	$\bar{14}$	13−	15
7	3	$\bar{14}$	38	45
8	3	$\bar{14}$	42	47

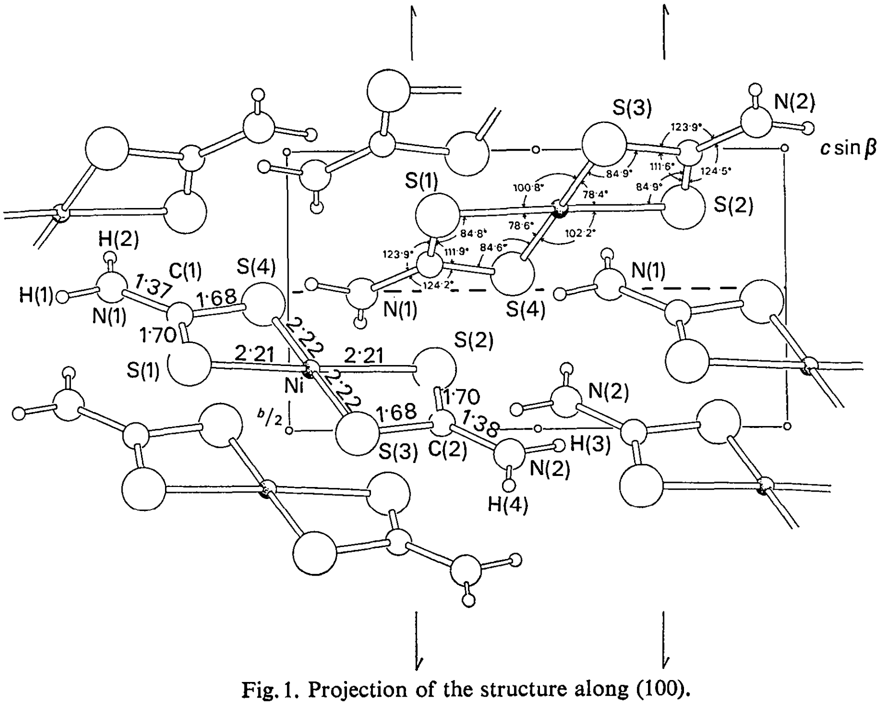


Fig. 1. Projection of the structure along (100).

distortion from planarity of sulphur atoms is tetrahedral in nature, the S(1) and S(2) atoms being at one side and S(3) and S(4) at the other side of the mean plane. Among the contacts involving the sulphur atoms (Fig. 2):

S(1)···S(3‴)	3·63 Å		
S(2)···N(2$^{\mathrm{iv}}$)	3·39	S(2)N(2$^{\mathrm{iv}}$)H(3$^{\mathrm{iv}}$)	23·4°
S(3)···N(1$^{\mathrm{v}}$)	3·38	S(3)N(1$^{\mathrm{v}}$)H(2$^{\mathrm{v}}$)	17·4
S(4)···N(2″)	3·42	S(4)N(2″)H(4″)	18·7

(for notation see Table 5) those concerning NH groups are consistent with the sum of hydrogen bond radii (Wallwork, 1962).

There are two long contacts roughly in the direction of the d_z orbitals of Ni

Ni···S(4′)	3·60 Å
Ni···N(2″)	3·48

so the coordination polyhedron is completed to a distorted and very elongated bipyramid (the angles formed by the directions Ni···S(4′) and Ni···N(2″) with the mean plane through S(1)S(2)S(3)S(4) are 80·0 and 70·1° respectively). The difference between these two contacts (0·12 Å) is not very dissimilar from the difference (0·2 Å) between the van der Waals radii of sulphur (1·7 Å, Nardelli, Braibanti & Fava, 1957; van der Helm, Lessor & Merritt, 1960; Nardelli, Fava Gasparri, Giraldi Battistini & Domiano, 1966) and of nitrogen (1·5 Å, Pauling, 1960) and this indicates that the two interactions are probably of the same kind. A relatively

Table 5. *Bond distances and angles*

The e.s.d.'s quoted in parentheses are in the units of the last place.

′ $\bar{x}, 1-y, \bar{z}$
″ $1-x, y-\frac{1}{2}, \frac{1}{2}-z$
‴ $1-x, 1-y, \bar{z}$
iv $1-x, 1-y, 1-z$
v $1+x, \frac{1}{2}-y, \frac{1}{2}+z$

(*a*) In the nickel coordination sphere

Ni–S(1)	2·207 (7) Å	S(1)–Ni–S(4)	78·6 (0·3)°
Ni–S(2)	2·210 (7)	S(1)–Ni–S(3)	100·8 (0·3)
Ni–S(3)	2·218 (15)	S(2)–Ni–S(3)	78·4 (0·2)
Ni–S(4)	2·224 (15)	S(2)–Ni–S(4)	102·2 (0·3)

Ni····S(4′)	3·599 (8) Å
Ni····N(2″)	3·485 (11)
Ni····H(4″)	2·856 (10)

(*b*) In the ligand molecules

C(1)–S(1)	1·70 (3) Å	C(2)–S(2)	1·70 (3) Å
C(1)–S(4)	1·68 (2)	C(2)–S(3)	1·68 (2)
C(1)–N(1)	1·37 (3)	C(2)–N(2)	1·38 (3)
S(1)–C(1)–N(1)	123·9 (1·9)°	S(2)–C(2)–N(2)	124·5 (1·9)°
S(1)–C(1)–S(4)	111·9 (1·1)	S(2)–C(2)–S(3)	111·6 (1·2)
S(4)–C(1)–N(1)	124·2 (2·1)	S(3)–C(2)–N(2)	123·9 (2·1)

(*c*) Angles in the coordination rings

S(1)–Ni —S(4)	78·6 (0·3)°	S(2)–Ni —S(3)	78·4 (0·2)°
C(1)–S(4)–Ni	84·6 (0·9)	C(2)–S(3)–Ni	84·9 (0·9)
S(4)–C(1)–S(1)	111·9 (1·1)	S(2)–C(2)–S(3)	111·6 (1·2)
C(1)–S(1)–Ni	84·8 (0·7)	C(2)–S(2)–Ni	84·9 (0·7)

Table 6. Ni–S *distances* (Å) *in planar* Ni(II) *complexes*

Four-membered rings		
Nickel bis(dithiocarbamate)	2·21 – 2·22	Present paper
Nickel bis(*N*,*N*′-diethyldithiocarbamate)	2·20 – 2·21	Bonamico, Dessy, Mariani, Vaciago & Zambonelli, 1965
Nickel bis(*N*,*N*′-di-n-propyldithiocarbamate)	2·20 – 2·21	Peyronel & Pignedoli, 1966
Nickel xanthate	2·23 – 2·24	Franzini, 1963
Mean value	2·216	
Five-membered rings		
Bis(thiosemicarbazidato)nickel	2·16	Cavalca, Nardelli & Fava, 1962
Bis(thiosemicarbazide)nickel sulphate (α form, *trans*)	2·16	Grønbaek & Rasmussen, 1962
Bis(thiosemicarbazide)nickel sulphate (β form, *cis*)	2·15	Grønbaek Hazell, 1966
Bis(thiosemicarbazide)nickel sulphate (β form, *trans*)	2·16	Grønbaek Hazell, 1966
Nickel bis(methylthiohydroxamate)	2·15	Sato, Nagata, Shiro & Koyama, 1966
Bis(maleonitriledithiolato)nickel	2·16	Eisenberg, Ibers, Clark & Gray, 1964
Mean value	2·157	

Table 7. *Best plane through the coordinated sulphur atoms*

Equation of the plane referred to orthogonal axes*
$-0{\cdot}5029x' + 0{\cdot}8527y' - 0{\cdot}1413z' = 2{\cdot}3823$.

	$\sigma_\perp$ (Å)†	Δ (Å)	$\Delta/\sigma_\perp$
S(1)	0·005	−0·030	6·0
S(2)	0·005	−0·025	5·0
S(3)	0·004	0·022	5·5
S(4)	0·005	0·026	5·2
Ni	(0·002)	(0·044)	(22·0)
$\Sigma(\Delta/\sigma_\perp)^2$			21·7
$\chi^2_{95\%}$			3·8

* The transformation matrix from monoclinic x,y,z, to orthogonal x',y',z' coordinates is:

$$\begin{pmatrix} 1 & 0 & \cos\beta \\ 0 & 1 & 0 \\ 0 & 0 & \sin\beta \end{pmatrix}$$

† $\sigma_\perp = \{m_1^2\sigma^2(x') + m_2^2\sigma^2(y') + m_3^2\sigma^2(z')\}^{\frac{1}{2}}$.

short approach to Ni also applies to the H(4″) which is at 2·85 Å from the metal atom; metal–hydrogen contacts of this type have already been observed also in copper(II) bis(*N*,*N*′-diethyldithiocarbamate) (Cu···H 2·86 Å, Bonamico, Dessy, Mugnoli, Vaciago & Zambonelli, 1965) in *trans*-diiodobis(dimethylphenylphosphine)palladium(II) (Pd···H 2·8 Å, Bailey, Jenkins, Mason & Shaw, 1965) and in dichlorotris(triphenylphosphine)ruthenium(II) (Ru···H 2·7 Å, Ibers, 1965).

Bond angles are not significantly different if compared in the two coordination rings and in nickel bis(diethyldithiocarbamate) (Bonamico, Dessy, Mariani, Vaciago & Zambonelli, 1965); they are probably determined not only by the geometry of the CS_2 group but also by the possibility these groups have of approaching each other and Ni in the planar arrangements imposed by the directive effects of bonding. In this connexion it is important to consider that the two S(1)···S(3) = 3·41 and S(2)···S(4) = 3·45 Å distances correspond to the shortest values hitherto observed for S···S contacts: *e.g.* 3·40 and 3·45 Å in bisthioureanickel thiocyanate (Nardelli, Fava Gasparri, Giraldi Battistini & Domiano, 1966), 3·43 Å in $K_2Co(NCS)_4.4H_2O$ (Ždanov & Zvonkova, 1950), 3·47 Å in rhodanine (van der Helm, Lessor & Merritt, 1960); according to these authors the van der Waals radius of sulphur should be 1·72–1·73 Å.

The four S–C distances (mean value 1·69 Å) are significantly shorter than the distance (1·76–1·78 Å)* which can be reasonably assumed for a S–C(sp^2) single

---

* These values have been found for the C–S distance in the following thiourea–metal complexes in which that bond is probably single in character: 1·76 Å in Cd*tu*$_2$(HCOO)$_2$ (Nardelli, Fava Gasparri & Boldrini, 1965), 1·76 Å in Ni*tu*$_2$(NCS)$_2$ (Nardelli, Fava Gasparri, Giraldi Battistini & Domiano, 1966), 1·76 Å in Cu *tu* SO_4 $2H_2O$ (Cavalca, Domiano, Fava Gasparri & Boldrini, 1967), 1·78 Å in Zn*tu*$_2$Cl$_2$ (Kunchur & Truter, 1958) (*tu* = thiourea).

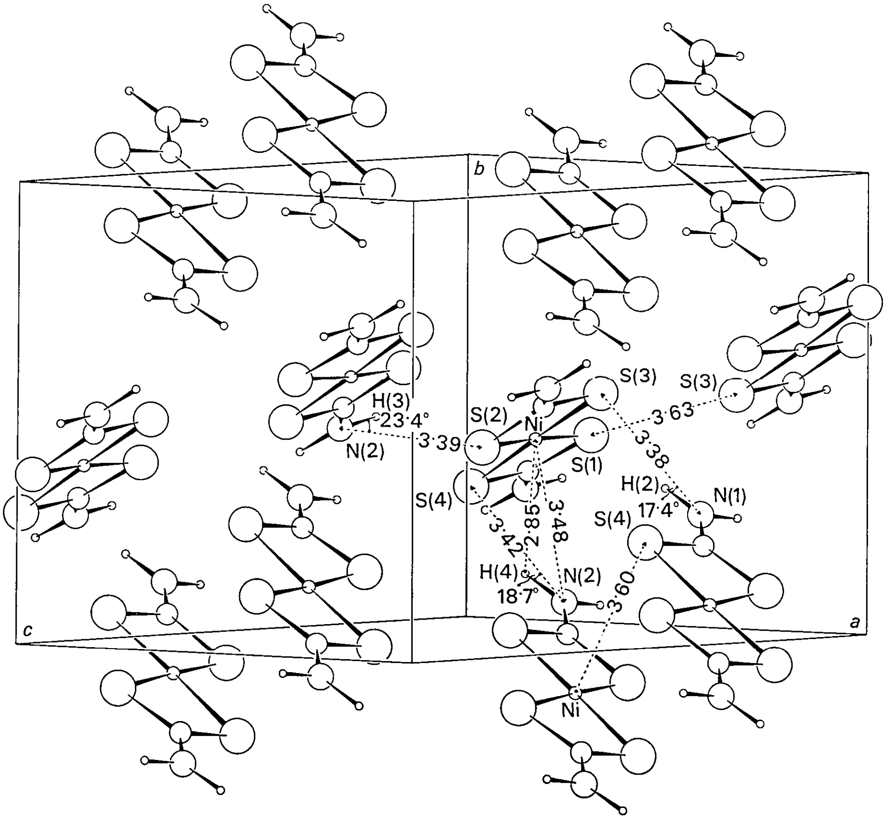
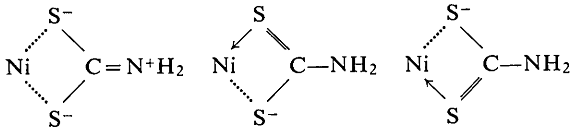


Fig. 2. Clinographic projection of the structure.

bond; they indicate, with the values for the C–N distances, that the canonical structures



postulated by Chatt, Duncanson & Venanzi (1956) on the basis of the infrared spectra, are all important in describing the bond configuration of the ground state of the ligand molecules.

All the calculations were performed on the Olivetti Elea 6001/S computer of the Centro di Calcolo Elettronico della Università di Parma. The authors are indebted to the Consiglio Nazionale delle Ricerche for financial support.

References

Ahmed, F. R. & Cruickshank, D. W. J. (1953). *Acta Cryst.* **6**, 385.
Alderman, P. R. H., Owston, P. G. & Rowe, J. M. (1962). *J. Chem. Soc.* p. 668.
Bailey, N. A., Jenkins, J. M., Mason, R. & Shaw, B. L. (1965). *Chem. Comm.* p. 237.
Berghuis, J., Haanappel, IJ. M., Potters, M., Loopstra, B. O., MacGillavry, C. H. & Veenendaal, A. L. (1955). *Acta Cryst.* **8**, 478.
Bonamico, M., Dessy, G., Mariani, C., Vaciago, A. & Zambonelli, L. (1965). *Acta Cryst.* **19**, 619.
Bonamico, M., Dessy, G., Mugnoli, A., Vaciago, A. & Zambonelli, L. (1965). *Acta Cryst.* **19**, 886.
Bonamico, M., Mazzone, G., Vaciago, A. & Zambonelli, L. (1965). *Acta Cryst.* **19**, 898.
Cavalca, L., Domiano, P., Fava Gasparri, G. & Boldrini, P. (1967). *Acta Cryst.* **22**, 878.
Cavalca, L., Nardelli, M. & Fava, G. (1962). *Acta Cryst.* **15**, 1139.
Chatt, J., Duncanson, L. A. & Venanzi, L. M. (1956). *Nature, Lond.* **177**, 1042.
Colapietro, M., Domenicano, A., Vaciago, A. & Zambonelli, L. (1966). *Acta Cryst.* **21**, A1 39.
Cruickshank, D. W. J. (1949). *Acta Cryst.* **2**, 65.
Cruickshank, D. W. J. (1956). *Acta Cryst.* **9**, 747.
Darlow, S. F. (1960). *Acta Cryst.* **13**, 683.
Darlow, S. F. & Cochran, W. (1961). *Acta Cryst.* **14**, 1250.
Dawson, B. (1960). *Acta Cryst.* **13**, 403.
Eisenberg, R., Ibers, J. A., Clark, R. J. H. & Gray, H. B. (1964). *J. Amer. Chem. Soc.* **86**, 113.
Franzini, M. (1963). *Z. Kristallogr.* **118**, 393.
Grønbaek, R. & Rasmussen, S. E. (1962). *Acta Chem. Scand.* **16**, 2325.
Grønbaek Hazell, R. (1966). *Acta Cryst.* **21**, A 142.
Helm, D. van der, Lessor, A. E. Jr. & Merritt, L. L. Jr. (1960). *Acta Cryst.* **13**, 1050.
Ibers, J. A. (1965). Abstr. Amer. Cryst. Assoc. Meeting, B10.
Klug, H. P. (1966). *Acta Cryst.* **21**, 536.
Kunchur, N. R. & Truter, M. R. (1958). *J. Chem. Soc.* p. 3478.
Lopez-Castro, A. & Truter, M. R. (1963). *J. Chem. Soc.* p. 1309.
Nardelli, M., Braibanti, A. & Fava, G. (1957). *Gazz. chim. ital.* **87**, 1209.
Nardelli, M. & Fava, G. (1960). *Ric. sci.* **30**, 898.
Nardelli, M., Fava Gasparri, G. & Boldrini, P. (1965). *Acta Cryst.* **18**, 618.
Nardelli, M., Fava Gasparri, G., Giraldi Battistini, G. & Domiano, P. (1966). *Acta Cryst.* **20**, 349.
Nardelli, M., Musatti, A., Domiano, P. & Andreetti, G. (1964). *Ric. sci.* **34**, II-A, 711.
Nardelli, M., Musatti, A., Domiano, P. & Andreetti, G. (1965). *Ric. sci.* **35**, II-A, 469, 477, 807.
Pauling, L. (1960). *The Nature of the Chemical Bond*, 3rd ed. Ithaca: Cornell Univ. Press.
Peyronel, G. (1941). *Z. Kristallogr.* **103**, 157.
Peyronel, G. & Pignedoli, A. (1966). *Acta Cryst.* **21**, A 156.
Phillips, D. C. (1956). *Acta Cryst.* **9**, 819.
Rollett, J. S. & Sparks, R. A. (1960). *Acta Cryst.* **13**, 273.
Sato, T., Nagata, K., Shiro, M. & Koyama, H. (1966). *Chem. Comm.* p. 192.
Shugam, E. A. & Agre, V. M. (1966). *Acta Cryst.* **21**, A 152.
Thomas, L. H. & Umeda, K. (1957). *J. Chem. Phys.* **26**, 293.
Wallwork, S. C. (1962). *Acta Cryst.* **15**, 758.
Ždanov, G. S. & Zvonkova, Z. V. (1950). *Zh. Fiz. Khim.* **24**, 1339; from *Structure Reports* **13**, 291.
Zvonkova, Z. V., Poveteva, Z. P., Vozzhennikov, V. M., Glushkova, V. P., Yakovenko, V. I. & Chvatkina, A. N. (1966). *Acta Cryst.* **21**, A 155.